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1. Your reference — NJE/DSH

2. Patent application number *(The Patent Office will fill in this part)* — 9919688.3

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

University College London
Gower Street
London
WC1E 6BT

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation — United Kingdom

4. Title of the invention — FABRICATION OF FABRY-PEROT POLYMER FILM SENSING INTERFEROMETERS

5. Name of your agent *(if you have one)* — ELKINGTON AND FIFE

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

ELKINGTON AND FIFE
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Patents ADP number *(if you know it)* — 67004

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Country	Priority application number *(if you know it)*	Date of Filing *(day/month/year)*

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Number of earlier application	Date of Filing *(day/month/year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer "Yes" if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d))*	yes

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Continuation sheets of this form	
Description	11
Claim(s)	3
Abstract	1
Drawing(s)	3

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Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	1
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11.

I/We request the grant of a patent on the basis of this application.

Signature Nick Ertl

Date 19 August 1999

12. Name and daytime telephone number of person to contact in the United Kingdom

Nick Ertl
01732 458 881

FABRICATION OF FABRY-PEROT POLYMER FILM SENSING INTERFEROMETERS

The present invention relates to an interferometer sensor and a method of manufacturing an interferometer sensor. In particular, the present invention relates to a method for forming a polymer film Fabry-Perot interferometer sensor.

A known interferometer comprises a polymer interferometer film, the deflection or compression of which, by a signal for analysis, modulates multiple reflections of an incident optical interrogation signal. For example, a known optical fibre interferometer using a polymer film comprises an optical fibre having a cleaved end and a polymer sensing film butted against the cleaved end. Two opposite faces of the polymer film provide the two reflecting surfaces of the interferometer. Light is introduced to the optical fibre and any external change that causes a variation in the optical thickness of the sensor film can be detected, since modulation of the thickness of the polymer film influences the output of the interferometer sensor. The external changes could include acoustic waves, quasi-static pressure and temperature variations ~~or~~ thermal waves caused by transient heating.

Conventionally, a disc of PET (polyethylene terepthalate) may be used as the polymer film. The disc is cut from a larger piece of the PET and adhered to the cleaved end of the optical fibre using a conventional adhesive agent. However, high uniformity in the thickness of the polymer film is required, and any irregularities in the surfaces of the PET or a any lack of uniformity in the thickness of the PET can adversely

affect the operation of the interferometer. The birefringence of the PET film also has adverse effects on the sensor operation. In addition, the process of cutting out and attaching the PET film to the cleaved end of the optical fibre is complex and time consuming.

Furthermore, the applicant has recognised that the use of an adhesive agent disposed between the inner surface of the PET disc and the cleaved end of the optical fibre can further affect the operation of the interferometer. In particular, due to its finite thickness, the adhesive agent can itself act as an additional interferometer film.

According to a first aspect of the present invention, there is provided a method of forming an interferometer film for an interferometer sensor comprising the step of forming a polymer layer of substantially uniform thickness directly on an interferometer substrate, the layer forming the interferometer film.

Since the interferometer film is formed directly onto the surface of the interferometer substrate, there is improved conformity between the two surfaces at the interface between the polymer layer and the substrate. Furthermore, improved uniformity in the thickness of the film can be achieved. Since no layer of adhesive is required to fix the interferometer film to the substrate, adverse interference effects from an adhesive layer are avoided.

The polymer may be formed by polymerising or by sputtering, or by any other method by which a polymer layer is caused to form directly on the surface of the

substrate.

For a polymerising process, the method may comprise, prior to the step of polymerising, the step of forming a gas of monomer particles in a first chamber at a first pressure and a first temperature and coupling the gas of monomer particles to a deposition chamber.

Preferably, the gas of monomer particles includes para-xylylene. Para-xylylene compounds are particularly effective in this application. They offer uniformity and completeness of coverage in addition to good physical, electrical, chemical, mechanical and barrier properties. Furthermore, no solvents are released during the coating process and the process is thus not affected by volatile organic compound (VOC) regulatory restrictions. In addition, the encapsulation provided by para-xylylene is excellent, being free of pin-holes in coatings as thin as $1\mu m$.

According to a second aspect of the present invention, there is provided an interferometer sensor comprising an interferometer substrate and a polymer film of substantially uniform thickness, in which the polymer film is formed directly on the interferometer substrate.

This sensor avoids interference affects caused by adhesive layers.

The invention also provides medical analysis equipment having an interferometer sensor assembly comprising:

an interferometer sensor of the invention;

an interrogation source to provide an interrogation signal to the sensor; and

a detector to detect signals received from the sensor.

Examples of the present invention will now be described in detail with reference to the accompanying drawings, in which:

Figure 1 shows an example of an inferometer including an optical fibre interferometer sensor according to the present invention;

Figure 2 shows an apparatus suitable for performing a method according to a first example of the present invention;

Figure 3 shows an apparatus suitable for performing a method according to a second example of the present invention.

Figure 4 shows an apparatus suitable for performing a method according to a third example of the present invention.

Interferometers are well-known for measuring physical parameters. This invention is particularly directed to a sensor and a method of manufacturing a sensor which operates according to the principles of a Fabry-Perot interferometer. Such a device may be used to study acoustic waves or thermal waves. For the purpose of explanation only, Figure 1 shows an example of an inferometer including an optical fibre interferometer sensor according to the present invention. Although an interferometer film is shown provided at the end of an optical fibre, the interferometer film may be provided simply on a support substrate with an interferometer interrogation signal being directed through free space to the

interferometer substrate.

The interferometer comprises an optical fibre 4 having a cleaved and polished end face 6. Butted against the end face 6 is a polymer film 8 having opposite parallel faces 10, 12 which are at least partially reflective to incident light from a given direction (from right to left in Figure 1). A light source and detector assembly 16 is provided to supply an optical interrogation signal 14 to the optical fibre 4. The face 12 is partially reflective so that some of the signal 14 is able to penetrate into the polymer film 8, and the face 10 may be 100% reflective. The reflectivities of the surfaces 10 and 12 may be obtained/controlled by providing a respective reflective coating to each of the surfaces 10 and 12 or by ensuring there is a refractive index mismatch between the optical fibre 4 and the polymer film 8 and between the polymer film and surrounding medium (for example water).

In use, the optical interrogation signal 14 is supplied to the optical fibre 4 and light is reflected from the two faces 10, 12 of the polymer film 8. An incident signal 2, for example containing information about a physical parameter of a sample being analysed, modulates the thickness of the film 8 and hence the optical phase difference between the light reflected from the two faces 10, 12. This produces a corresponding intensity modulation of the light reflected from the film 8. As such, information about the sample can be obtained.

In conventional optical fibre polymer film interferometers, an adhesive agent is used to secure film 8 against the cleaved end 6 of the optical fibre 4. The applicant has

recognised that this is undesirable since, as described above, the adhesive has a finite thickness and can act as a reflective film itself thereby introducing undesired interference to the detected optical signals. In accordance with the invention the film is formed directly onto the interferometer substrate, for example the end of an optical fibre, to form a substantially uniform thickness layer of the polymer substance on the substrate. No adhesive is required and as such the output from the interferometer sensor is improved. Furthermore, a wide choice of polymer materials is available, as the polymerised layer does not need to withstand removal from the substrate on which it is polymerised for subsequent attachment to another substrate. Therefore, a polymer can be selected which does not exhibit birefringence problems, and the complexity of manufacture is reduced.

Figure 2 shows a first example of an apparatus suitable for performing the method of the present invention. The apparatus described is for forming an interferometer sensor using a parylene polymerisation process. The apparatus has an inlet chamber 18, a pyrolysis chamber 20 and a deposition chamber 22 connected by hermetically sealed tubing 24. An optical fibre 26, having an end face to be coated by the parylene, is introduced to the deposition chamber 22 via inlet valve 28. Areas of the optical fibre that are to remain free of coating are masked since the active parylene monomer will polymerise on any available surface.

In use, a dimer parylene precursor is introduced into inlet chamber 18 via tubing where it is vaporised at approximately 150°C and in a 100Pa vacuum. The vaporised monomer continues via tubing 24 to the pyrolysis chamber 20 where it is heated to

a temperature of approximately 680°C in a 50Pa vacuum.

The highly active parylene monomer gas continues via tubing 25 to the deposition chamber 22. The deposition chamber is typically at ambient room temperature and has an internal pressure of 10Pa. The optical fibre 26 is placed in the deposition chamber 22 with an exposed surface onto which the parylene monomer can polymerise.

The monomer simultaneously condenses, adsorbs and polymerises on all available surfaces to produce a high molecular-weight polymer coating. Due to the chemical properties of para-xylylene and the polymerisation mechanism, the coating formed is conformal and has uniform thickness. In particular, the parylene deposition process does not entrap air since the process is carried out in an effective vacuum. The optical fibre is then removed and demasked and the coating thickness is checked.

There are three common forms of the parylene polymer, parylene C, parylene N and parylene D. Typically, the parylene coating grows at approximately 0.2μm per minute for parylene C and a slower rate for parylene N. The polymers each have high hydrophobicity and as such are particularly useful as sensors for medical probe applications.

It is important that the optical fibre being coated is clean and surface contaminants such as oils and ions are removed prior to the coating process. To perform the cleaning process prior to the coating process, a multi-molecular layer of an organo-

silane may be applied to pretreat the parts of the optical fibre that are to be coated. This functions as an adhesion promoter, allowing the polymers to be applied to virtually any vacuum stable material.

The parylene polymerisation process described above involves simultaneous condensation, adsorption and polymerisation of the highly active monomer gas on all available surfaces of the exposed substrate. Other suitable monomer gasses can also be deposited and polymerised on the substrate in this manner. The example described of parylene is in no way intended to be limiting and any other suitable substance could also be used to form the interferometer film.

Alternatively, polymerisation from a plasma may be performed. Figure 3 shows an apparatus suitable for performing a deposition method according to a second example of the present invention. In this case, a plasma polymerisation process is used to form the interferometer layer on the cleaved end of the optical fibre 26. In this example, a plasma of CF_4 is used to form the polymer layer of PTFE. Any suitable monomer that polymerises to form a polymer having the required optical, mechanical and physical properties could be used. For example methylmethacrylate, ethylene or styrene could each be used to form the polymer film.

The apparatus comprises a first chamber 30 coupled to second deposition chamber 32 via hermetically sealed tubing 34. An RF-plasma excitation electrode 36 excites a gas of monomer particles into a plasma state which is then used in a plasma polymerisation process to form a layer of a polymer derived from the CF_4 monomer

on the exposed surfaces of the optical fibre 26.

A monomer gas including CF_4 is generated in chamber 30 and coupled to deposition chamber 32. The introduction of the monomer gas raises the pressure in the evacuated deposition chamber 32 to approximately 17 Pa. The CF_4 is then partially ionised using electrode 36 to generate the plasma. A plasma polymerisation process then occurs in which a layer of $(CF_4)_n$ is formed on the exposed surfaces of optical fibre 26. The optical fibre is removed and demasked and the thickness of the coating may be checked. In the example of Figure 3, the end of the fibre 26 is introduced through a port 28 into the deposition chamber 32.

As in the parylene polymerisation process described above, it is important that the optical fibre being coated is clean and surface contaminants such as oils and ions are removed prior to the coating process. This may be done in a conventional manner. Again, since the interferometer layer is polymerised directly onto the surface of the cleaved end of the optical fibre, the uniformity achieved in the thickness of the film and the conformity at the polymer/fibre interface is improved.

Figure 4 shows a further example of an apparatus suitable for forming the interferometer film according to the present invention. In this case, RF sputtering is used to deposit an interferometer film directly onto a substrate in the form of a transparent support plate mounted in a deposition chamber. The apparatus includes an evacuated chamber 38 and an RF generator power source 40. A first electrode 44 is coupled to the RF power source 40 and a second electrode 46 is coupled to earth.

A spacing is maintained between the electrodes 44 and 46 and a bulk polymer sample 42 is introduced adjacent to first electrode 44. A substrate 45 to be coated is introduced adjacent to second electrode 46.

In use, a gas is introduced to the evacuated chamber 38 via inlet valve 39 and a voltage is applied between electrodes 44 and 46. As the applied voltage increases above a threshold value ionisation of the gas occurs and causes a glow discharge between the electrodes 44 and 46 and therefore ion bombardment of the sample 42. The ion bombardment causes the removal of molecules from the bulk polymer sample 42 which then condense on the substrate 45 creating a conformal uniform film. In the case of a PTFE polymer film being formed, low weight fragments such as C_2F_4 form a substantial proportion of the species arriving at the substrate for recombination to form a polymer. The gas used to generate the glow discharge is preferably an inert gas such as Argon, although obvious alternatives may of course also be used.

As with polymerisation, there is no liquid phase involved in the process of forming the interferometer film, and therefore the coating formed is substantially uniform and conforms to the substrate surface.

Although examples have been given of three possible processes, those skilled in the art will appreciate that various alternative processes are possible and it is also apparent that a range of polymers may be selected. The choice of a particular polymer/monomer will depend on desired optical, physical and mechanical properties and deposited thickness of the layer. For example, any polymers having the required

values for transparency, water absorption, Young's Modulus, thermal expansivity and thermal stability and being non-birefringent would be suitable. The deposition techniques described above also provide good adhesion of the inferometer film to the substrate.

As examples, a sensor of the invention may be used for analysing ultrasonic acoustic waves, for medical imaging applications, non-destructive testing of materials, characterisation of industrial ultrasonic processes (for example ultrasonic cleaning or sterilisation processes) or analysing ultrasound source outputs. The sensor may also be used for analysing quasi-static pressure, for example for intra-arterial blood pressure measurement or for pressure measurement in hydraulic systems. The sensor may also be used for analysing quasi-static temperature, for example for temperature measurement during heating of biological tissue. Detection of thermal waves is also possible for biomedical photothermal techniques. The possible uses of a Fabry-Perot interferometer will be apparent to those skilled in the art.

CLAIMS

1. A method of forming an interferometer film for an interferometer sensor comprising the step of forming a polymer layer of substantially uniform thickness directly on an interferometer substrate, the layer forming the interferometer film.

2. A method as claimed in claim 1, in which the forming comprises polymerising a substance onto the substrate.

3. A method according to claim 2, further comprising, prior to the polymerisation, the step of forming a gas of monomer particles in a first chamber at a first pressure and a first temperature and coupling the gas of monomer particles to a deposition chamber.

4. A method according to claim 3, wherein the substrate is placed in the deposition chamber and, at a second pressure and second temperature, monomer particles polymerise on the substrate.

5. A method according to claim 3 or 4, in which the gas of monomer particles includes a plasma of an organic vapour.

6. A method according to claim 3 or 4, in which the gas of monomer particles includes a para-xylylene.

7. A method according to any preceding claim, in which the forming is carried out under vacuum conditions.

8. A method according to claim 1, in which the forming comprises sputtering the polymer onto the substrate.

9. A method according to any preceding claim, in which the substrate is the cleaved end of an optical fibre.

10. A method substantially as described with reference to any one of Figures 2 to 4 of the accompanying drawings.

11. An interferometer sensor comprising an interferometer substrate and a polymer film of substantially uniform thickness, in which the polymer film is formed directly on the interferometer substrate.

12. A sensor according to claim 11, in which the polymer film is formed by a method according to any of claims 1 to 9.

13. A sensor substantially as shown in and/or described with reference to any of Figures 1 to 4 of the accompanying drawings.

14. Medical analysis equipment having an interferometer sensor assembly comprising:

an interferometer sensor according to any one of claims 11 to 13;

an interrogation source to provide an interrogation signal to the sensor; and

a detector to detect signals received from the sensor.

ABSTRACT

FABRICATION OF FABRY-PEROT POLYMER FILM SENSING INTERFEROMETERS

A method of forming an interferometer film for an interferometer sensor comprises forming a polymer layer (8) of substantially uniform thickness directly on an interferometer substrate (4;45), the layer forming the interferometer film. Since the interferometer film (8) is formed directly onto the surface of the interferometer substrate, there is improved conformity between the two surfaces at the interface between the polymer layer and the substrate and improved uniformity in the thickness of the film.

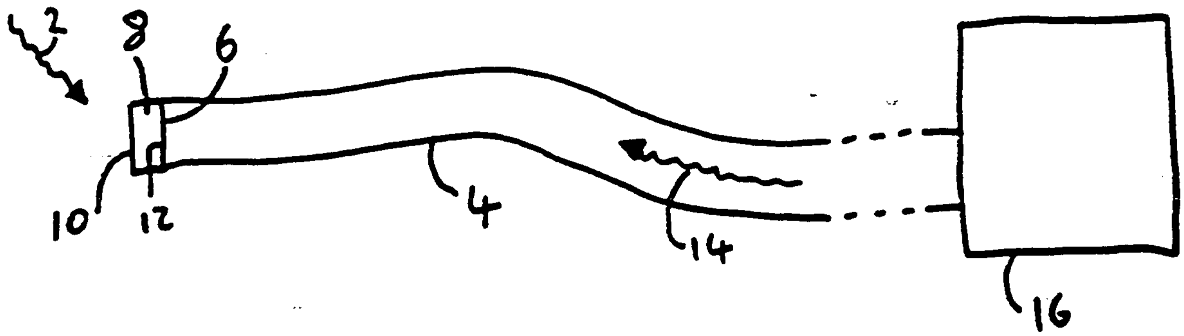

Fig. 1

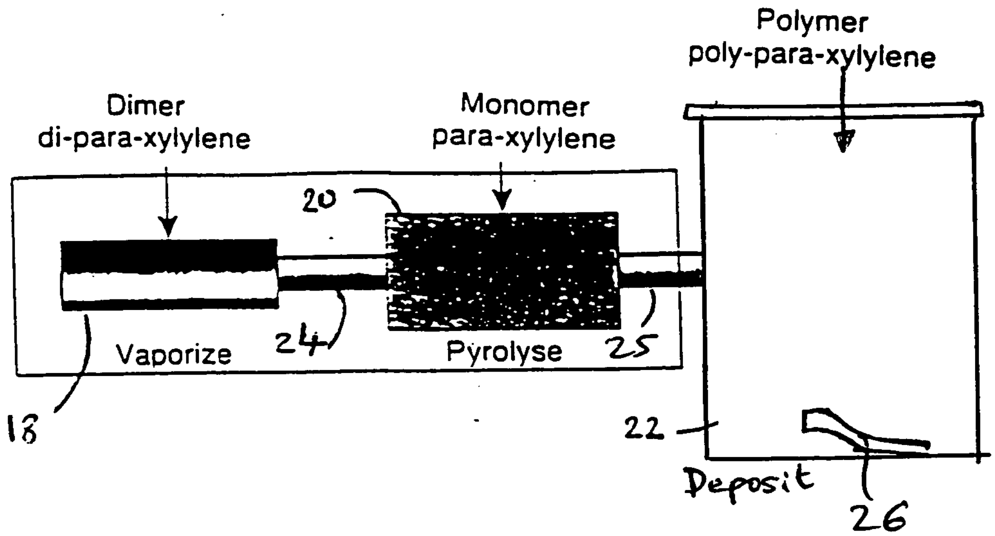
Polymer
poly-para-xylylene
Dimer
di-para-xylylene
Monomer
para-xylylene
20
Vaporize
24
Pyrolyse
25
18
22
Deposit
26

Fig 2

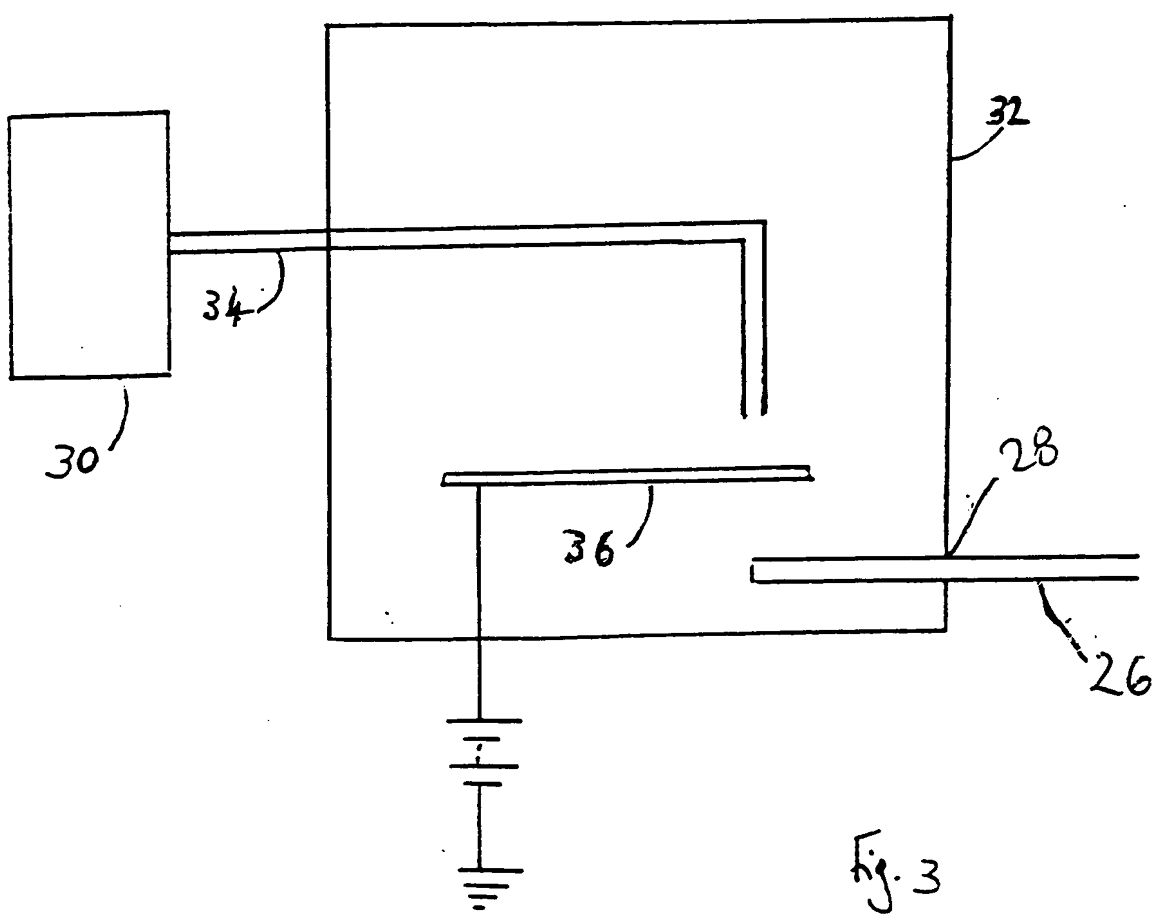
32
34
30
28
36
26

Fig. 3

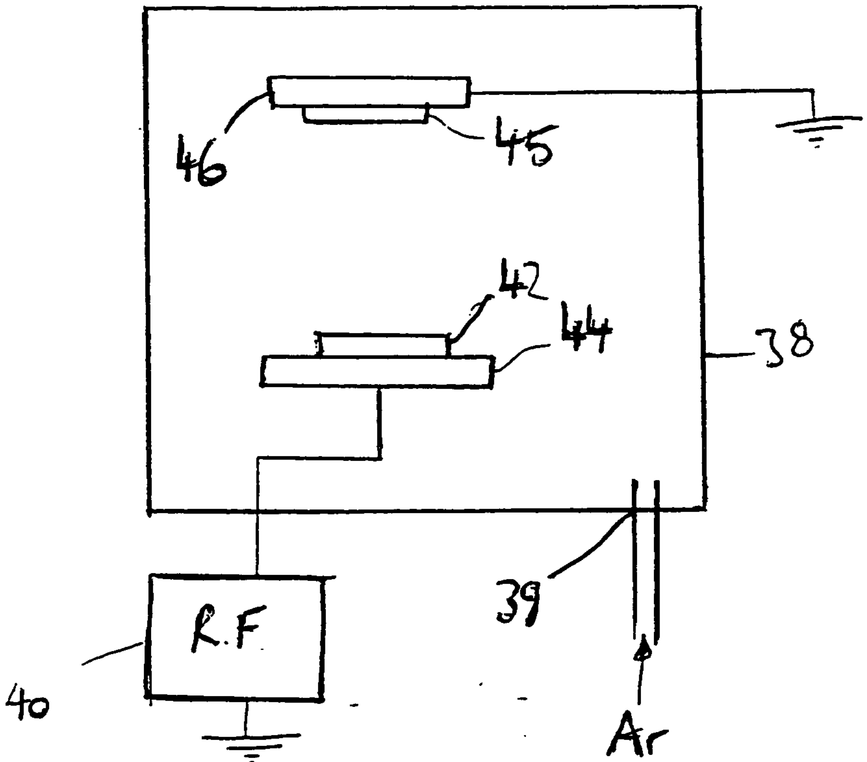

FIG 4

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